

should be close to the sampler during testing.

(c) We recommend the use of isotope dilution techniques, including the use of isotopically labeled surrogate, internal, alternate, and injection standards.

(d) If your target analytes degrade when exposed to ultraviolet radiation, such as nitropolynuclear aromatic hydrocarbons (nPAHs), perform these procedures in the dark or with ultraviolet filters installed over the lights.

(e) The following definitions and abbreviations apply for SVOC measurements:

(1) *Soxhlet extraction* means the extraction method invented by Franz von Soxhlet, in which the sample is placed in a thimble and rinsed repeatedly with a recycle of the extraction solvent.

(2) *XAD-2* means a hydrophobic crosslinked polystyrene copolymer resin adsorbent known commercially as Amberlite® XAD®-2, or an equivalent adsorbent like XAD-4.

(3) *Semi-volatile organic compound (SVOC)* means an organic compound that is sufficiently volatile to exist in vapor form in engine exhaust, but that readily condenses to liquid or solid form under atmospheric conditions. Most SVOCs have at least 14 carbon atoms per molecule or they have a boiling point between (240 and 400) °C. SVOCs include dioxin, quinone, and nitro-PAH compounds. They may be a natural byproduct of combustion or they may be created post-combustion. Note that SVOCs may be included in measured values of hydrocarbons and/or PM using the procedures specified in this part.

(4) *Kuderna-Danish concentrator* means laboratory glassware known by this name that consists of an air-cooled condenser on top of an extraction bulb.

(5) *Dean-Stark trap* means laboratory glassware known by this name that uses a reflux condenser to collect water from samples extracted under reflux.

(6) *PUF* means polyurethane foam.

(7) *Isotopically labeled* means relating to a compound in which either all the hydrogen atoms are replaced with the atomic isotope hydrogen-2 (deuterium) or one of the carbon atoms at a defined position in the molecule is replaced with the atomic isotope carbon-13.

§ 1065.1105 Sampling system design.

(a) *General.* We recommend that you design your SVOC batch sampler to extract sample from undiluted emissions to maximize the sampled SVOC quantity. To the extent practical, adjust sampling times based on the emission rate of target analytes from the engine to obtain analyte concentrations above the detection limit. In some instances you may need to run repeat test cycles without replacing the sample media or disassembling the batch sampler.

(b) *Sample probe, transfer lines, and sample media holder design and construction.* The sampling system should consist of a sample probe, transfer line, PM filter holder, cooling coil, sorbent module, and condensate trap. Construct sample probes, transfer lines, and sample media holders that have inside surfaces of nickel, titanium or another nonreactive material capable of withstanding raw exhaust gas temperatures. Seal all joints in the hot zone of the system with gaskets made of non-reactive material similar to that of the sampling system components. You may use teflon gaskets in the cold zone. We recommend locating all components as close to probes as practical to shorten sampling system length and minimize the surface exposed to engine exhaust.

(c) *Sample system configuration.* This paragraph (c) specifies the components necessary to collect SVOC samples, along with our recommended design parameters. Where you do not follow our recommendations, use good engineering judgment to design your sampling system so it does not result in loss of SVOC during sampling. The sampling system should contain the following components in series in the order listed:

(1) Use a sample probe similar to the PM sample probe specified in subpart B of this part.

(2) Use a PM filter holder similar to the holder specified in subpart B of this part, although you will likely need to use a larger size to accommodate the high sample flow rates. We recommend using a 110 mm filter for testing spark ignition engines or engines that utilize exhaust aftertreatment for PM removal and a 293 mm filter for other engines. If you are not analyzing separately for SVOCs in gas and particle

phases, you do not have to control the temperature of the filter holder. Note that this differs from normal PM sampling procedures, which maintain the filter at a much lower temperature to capture a significant fraction of exhaust SVOC on the filter. In this method, SVOCs that pass through the filter will be collected on the downstream sorbent module. If you are collecting SVOCs in gas and particle phases, control your filter face temperature according to §1065.140(e)(4).

(3) Use good engineering judgment to design a cooling coil that will drop the sample temperature to approximately 5 °C. Note that downstream of the cooling coil, the sample will be a mixture of vapor phase hydrocarbons in CO₂, air, and a primarily aqueous liquid phase.

(4) Use a hydrophobic sorbent in a sealed sorbent module. Note that this sorbent module is intended to be the final stage for collecting the SVOC sample and should be sized accordingly. We recommend sizing the module to hold 40 g of XAD-2 along with PUF plugs at either end of the module, noting that you may vary the mass of XAD used for testing based on the anticipated SVOC emission rate.

(5) Include a condensate trap to separate the aqueous liquid phase from the gas stream. We recommend using a peristaltic pump to remove water from the condensate trap over the course of the test to prevent build-up of the condensate. Note that for some tests it may be appropriate to collect this water for analysis.

(d) *Sampler flow control.* For testing using the recommended filter and sorbent module sizes, we recommend targeting an average sample flow rate of 70 liters per minute to maximize SVOC collection. The sampler must be designed to maintain proportional sampling throughout the test. Verify proportional sampling after an emission test as described in §1065.545.

(e) *Water bath.* Design the sample system with a water bath in which the cooling coil, sorbent module, and condensate trap will be submerged. Use a heat exchanger or ice to maintain the bath temperature at (3 to 7) °C.

§1065.1107 Sample media and sample system preparation; sample system assembly.

This section describes the appropriate types of sample media and the cleaning procedure required to prepare the media and wetted sample surfaces for sampling.

(a) *Sample media.* The sampling system uses two types of sample media in series: The first to simultaneously capture the PM and associated particle phase SVOCs, and a second to capture SVOCs that remain in the gas phase, as follows:

(1) For capturing PM, we recommend using pure quartz filters with no binder. Select the filter diameter to minimize filter change intervals, accounting for the expected PM emission rate, sample flow rate, and number of repeat tests. Note that when repeating test cycles to increase sample mass, you may replace the filter without replacing the sorbent or otherwise disassembling the batch sampler. In those cases, include all filters in the extraction.

(2) For capturing gaseous SVOCs, utilize XAD-2 resin contained between two PUF plugs.

(b) *Sample media and sampler preparation.* Prepare pre-cleaned PM filters and pre-cleaned PUF plugs/XAD-2 as needed. Store sample media in containers protected from light and ambient air if you do not use them immediately after cleaning.

(1) Pre-clean the filters via Soxhlet extraction with methylene chloride for 24 hours and dry over dry nitrogen in a low-temperature vacuum oven.

(2) Pre-clean PUF and XAD-2 with a series of Soxhlet extractions: 8 hours with water, 22 hours with methanol, 22 hours with methylene chloride, and 22 hours with toluene, followed by drying with nitrogen.

(3) Clean sampler components, including the probe, filter holder, condenser, sorbent module, and condensate collection vessel by rinsing three times with methylene chloride and then three times with toluene. Prepare pre-cleaned aluminum foil for capping the probe inlet of the sampler after the sampling system has been assembled.

(c) *Sorbent spiking.* Use good engineering judgment to verify the extent to which your extraction methods recover